

MICROCHEMICAL METHODS

Report on Microchemical Methods*

By C. L. OGG (Eastern Regional Research Laboratory,[†] Philadelphia 18, Pa.)

The second collaborative study on fluorine was conducted during the year. In this study the collaborators were supplied a detailed procedure which was based on the results of last year's work. The results show the method to be sufficiently reliable to warrant recommending it for adoption as first action.

Work has been conducted in the Referee's laboratory during the year on a micro method for determining molecular weight which uses thermistors to measure small differences in temperature between drops of solvent and solution suspended in an atmosphere saturated with solvent. The method is similar to that used by the collaborator who obtained the best results in last year's study. This method looks quite promising and is being tried by a number of laboratories throughout the country. Because there is no commercially available apparatus, each laboratory has had to construct its own,

and consequently there probably are no two apparatuses exactly the same. The principle, however, is the same in each case and the techniques are similar. A continuation of this study seems feasible.

The search for a modified Kjeldahl method which would be satisfactory for the analysis of materials containing N-N or N-O was not successful and this work should be abandoned.

It is recommended¹—

(1) That the method for fluorine tested this year be adopted as first action.

(2) That the study of methods for the determination of oxygen be continued.

(3) That the study of methods for the determination of molecular weight be continued.

(4) That the study of Kjeldahl methods, modified to determine nitrogen in N-N and N-O linkages, be discontinued.

(5) That the first action micro Kjeldahl method, 38.009–38.011, be made official.

* Presented as the report of the General Referee on Microchemical Methods at the Seventy-fourth Annual Meeting of the Association of Official Agricultural Chemists, Oct. 10–12, 1960, Washington, D.C.

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¹ These recommendations were approved by Subcommittee C and were adopted by the Association. See *This Journal* 44, 70 (1961).

Microanalytical Determination of Fluorine*

By AL STEYERMARK (Hoffmann-La Roche Inc., Nutley, N.J.)

In last year's collaborative study (7) collaborators were asked to analyze three samples by whatever methods they were using in their respective laboratories. The majority of the collaborators used Schöniger oxygen flask combustion (5, 8) for destroying organic material and ended the deter-

mination volumetrically, using thorium nitrate as the titrant. The procedure (8, 12) in use in the author's laboratory for the past two years included this method; consequently it was submitted to the collaborators for this year's study.

The collaborators were requested to analyze the following: (a) 5-fluorouracil; (b) *m*-trifluoromethyl benzoic acid; (c) Teflon¹; (d) 5-fluorouracil to which the analyst

* Presented as the report of the Associate Referee on Microchemical Methods for Fluorine at the Seventy-fourth Annual Meeting of the Association of Official Agricultural Chemists, Oct. 10–12, 1960, Washington, D.C.

¹ E. I. DuPont de Nemours and Co., Inc.

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